

PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

Separation of Mixtures of Fats and Fatty Acids

We, CHEMETRON CORPORATION, of 840 North Michigan Avenue, Chicago 11, State of Illinois, United States of America, a corporation organized and existing under the laws of the State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an improved process which may be employed to separate either mixed fatty acids or mixed triglycerides into fractions.

Natural occurring triglycerides, whether of animal or of vegetable origin, in most instances contain both unsaturated and saturated fatty acid radicals. Accordingly, when such triglycerides are subjected to separating processes to produce free fatty acids, mixtures of saturated and unsaturated fatty acids result. With fatty acids of approximately the same number of carbon atoms in the chain the saturated acids have higher melting points than the unsaturated fatty acids. Because of this difference in melting points and other different properties between unsaturated and saturated acids it is frequently desired to separate unsaturated acids from saturated acids in order to obtain product fractions of greater utility.

In the past the most commonly used method of separating unsaturated from saturated acids on a commercial scale was the pressing method. This method consists of cooling a molten mixture of acids without agitation until the mixture is partially solidified. The resulting mass is then enclosed in a porous bag or other filtering medium and subjected to mechanical pressure to force the liquid fraction through the filter medium. This method, is, at best, relatively

inefficient in effecting a sharp separation, and in order to obtain fractions of desired commercial purity it is usually necessary to resort to three such cooling and pressing operations in order to achieve a high purity saturated acid fraction. Hence the expression "triple pressed" has been applied as the conventional term for identifying stearic or other saturated acids of high purity. The pressing method, however, is generally unsatisfactory for it is quite messy to carry out and involves considerable manual labor. Accordingly, repeated attempts have been made to effect separation of fatty acids on a continuous basis by selective crystallization from a solvent medium. The generally used solvents have been petroleum hydrocarbons such as propane and hexane or non-hydrocarbon solvents such as acetone and methanol. A chief difficulty with the prior solvent fractionation processes is that the crystals formed when the solvent fatty acid mixture is chilled are very difficult to filter. Moreover, these crystals include relatively large amounts of unsaturated material, and in order to obtain high purity products successive stages of crystallization must be utilized. Moreover, high ratios of solvent to fatty material must be employed necessitating extensive solvent recovery systems and unless slow chilling with slow crystal growth is employed, a poor separation is obtained with the result that very large refrigerated chilling devices have been utilized.

All of the foregoing difficulties are also experienced, perhaps to an even greater degree, when attempting to separate unsaturated and saturated triglycerides such as the separation of natural lard into lard oil and a solid saturated triglyceride fraction.

One object of this invention is to provide

an improved continuous process capable of effecting, in a single crystallization step, a relatively sharp separation of saturated from unsaturated fatty acids or fatty triglycerides.

5 In the accompanying drawing, Fig. 1 is a process flow diagram of one example of a continuous plant for carrying out the process of this invention.

10 We have discovered that, if there is added to a mixture of saturated and unsaturated fats or fatty acids dissolved in a solvent fractional percentage amount of a class of substances which have the effect of promoting or controlling the crystal growth of the saturated fatty acids or triglycerides and that the thus formed solution is then chilled to a temperature slightly below the precipitation temperature of the saturated material in the solvent, a sharp separation 20 of the saturated fatty acids or triglycerides which precipitates in the form of readily filterable crystals may be obtained. Moreover, we have discovered that by utilizing this technique the chilling may be effected very rapidly and a relatively low solvent ratio may be utilized.

30 The crystals formed by the above described procedure appear upon microscopic examination to have a structure much like grains of sand and are generally in the form of irregular granular or spherical particles whereas if the crystals promoting substance is not added, the crystals which are normally formed exhibit a slimy plate-like structure and are extremely difficult, if not impossible, to subject to efficient filtration and washing operation. It appears that the saturated fatty acid chains of the crystal promoter which extend outward from the kernel become associated with molecules of triglyceride or fatty acid, so that each molecule of crystal promoter is associated with several molecules of triglyceride or fatty acid in the solution. Crystallization of the triglyceride 45 or fatty acid occurs during this association, with the result that the crystals which are formed are approximately equidimensional rather than in the form of flat plates as they normally occur. The equidimensional crystal form is particularly desirable because the crystals are easy to filter and the suspension of the crystals in solvent is fluid rather than viscous as in the normal situation where crystal promoters are not used.

50 A variety of different substances have been found to have the desired crystal promoting effect. All of the substances which we have found to have such effect, however, exhibit the following characteristics: They are compounds having at least four to 700 saturated fatty acid radicals, each radical having from sixteen to twenty-two carbon atoms; the fatty acid radicals may diverge either from a central nucleus or from an aliphatic chain. It is preferred that the saturated fatty acid

radical be identical with the radical of the fatty acid or triglyceride which is the desired product to be crystallized. The ratio by weight of solvent to fatty acid or triglyceride can be within the range of 1 to 1 70 and 4 to 1. It is preferred that the ratio by weight of solvent to fatty acid or triglyceride be within the range of 1 to 1 and 2 to 1.

75 From the variety of compounds tested, it appears that the size or configuration of the central nucleus of the molecule has little or no effect upon the effectiveness of crystal promoters of this type. Rather, it is important only that the kernel or nucleus be capable of attaching branches such as the saturated fatty acid radical chain in such a manner that they extend from the kernel or nucleus in more than one plane.

80 One of the preferred types of crystal promoter used in this invention comprises polyvinyl esters of fatty acids wherein the fatty acids contain 16 to 22 carbon atoms. For instance, polyvinyl stearate having on the average about 50 ester groups per molecule is an extremely effective crystal promoter. However, polyvinyl stearate of higher molecular weight is also effective. For instance, polyvinyl stearate of an average molecular weight of 90,000 (about 300 ester units per molecule) has been shown to be extremely effective. Another specimen of polyvinyl stearate having an average molecular weight of about 200,000 is effective. This substance has approximately 700 ester 90 linkages per molecule.

95 Polyvinyl esters of fatty acids, such as oleic and related unsaturated acids also form effective crystal promoters. For instance, a polyvinyl ester of lard fatty acids having approximately 50 ester groups per molecule is as effective as polyvinyl stearate of approximately the same molecular weight. Polyvinyl esters made from soybean oil fatty acid (containing not more than about 15% saturated higher fatty acids) have approximately the same effectiveness as crystal promoters as polyvinyl stearate of the same molecular weight. On the other hand, a polyvinyl ester made from tall oil fatty acids which consists almost entirely of unsaturated acids was ineffective, which seems to indicate that it is necessary to have some saturated fatty acid radicals containing 16 to 22 carbon atoms present, but that the proportion need 100 not be more than 10 or 15%.

105 Mixed polyvinyl esters of acetic and fatty acids, such as polyvinyl acetate palmitate and polyvinyl acetate stearate, are effective crystallization promoters even though the higher fatty acid radicals amount to only five or six per molecule.

110 The esters which are effective as crystal promoters have melting points which vary over a fairly wide range without adversely 120 125 130

influencing the effectiveness of the materials. For instance, polyvinyl stearate with a melting point of about 44° C. has good effectiveness while polyvinyl stearates containing mixed acids of lard or coconut oil are also effective even though they do not crystallize at temperatures as low as -60° C. The molecular weight of the polyvinyl ester does not seem to have any great influence on the effectiveness of the crystal promoters as shown by the fact that polyvinyl fatty acid esters with molecular weights from about 1500 to about 700,000 are effective without any significant change in effectiveness.

Fatty acid esters of polyhydric substances, such as sugars and sugar derivatives, are generally effective as crystal promoters so long as they contain 4 or more fatty acid ester groups wherein the fatty acid contains 16 to 22 carbon atoms. For instance, sucrose with most of its hydroxyl groups esterified with stearic acid is quite effective whereas sucrose dipalmitate is ineffective at 0.2% concentration. This indicates that the number of fatty acid groups per molecule is important. Methyl glucoside, which contains four hydroxyl radicals, produces a moderately effective crystal promoter when fully esterified with stearic acid.

Aluminum salts of fatty acids, such as aluminum stearate, are effective when suspended in oils or fats before being added to the solution to be crystallized, whereas ordinary aluminum stearate without presolution in oils or fats is ineffective. For instance, 9 parts of melted stearic acid are admixed with 1 part of aluminum stearate and the mixture allowed to cool and solidify. This product after being disintegrated into chips is highly effective in inducing crystallization of saturated fatty acids from solution containing the same. Likewise, the aluminum salt of tallow acids when dissolved in an excess of solidified tallow acids and disintegrated is also effective for crystallization of fatty acids.

Polyamide resins derived from fatty acids and alkylenediamines are effective in promoting crystallization of fatty acids. These resins are condensation polymers of dimerized and trimerized unsaturated fatty acids from vegetable oils, and of aromatic or aliphatic polyamines. For instance, the methyl esters of soybean fatty acids (linoleic and linolenic acids but containing about 15% saturated fatty acids) are suitably polymerized and then condensed with an equivalent amount of ethylenediamine at 150-225° C. in an inert atmosphere, the reaction being completed under vacuum at 200-225° C. Similar polyamide resins are produced from linoleic, linolenic and oleostearic acids found in linseed, tung and dehydrated castor oils. Polyamines which

are suitable include ethylenediamine, diethylenetriamine, hexamethylenediamine, triethylenetetramine, propylenediamine and butylenediamine. Such polyamide resins have molecular weights of 3000-5000 and are soluble in common organic solvents, such as isopropyl alcohol, n-butyl alcohol, chloroform, cyclohexanol and pyridine. The preparation and properties of such polyamide resins are described by Cowan et al, *Oil & Soap*, pp. 769 et seq. (1941); Cowan et al, *Oil & Soap*, pp. 101-107 (1941); Falkenberg et al, *Oil & Soap*, pp. 143-147 (1945); and Renfrew et al, *Industrial and Engineering Chemistry*, Vol. 46, pp. 2226-2231 (1954).

Substances which have been tested and which clearly do not produce the desired result are as follows: natural waxes, shellac, rosin, various triglycerides, polyethylene, dimerized fatty acids, various amines such as octylamine, ethylene diamine, tetraethylene pentamine, butyl stearate, methoxyethyl stearate, sorbitan tristearate, polyvinyl esters of the following: unhydrogenated soybean oil acids, tall oil fatty acids and lauric acid.

The minimum amount of modifier required depends upon the particular modifier and in most cases appears to be approximately one tenth of one per cent by weight of the fatty material. In most instances two tenths of one per cent by weight is the preferred amount, particularly when the preferred modifier as described in Example 1 below is utilized. Some of the materials listed above as being satisfactory are substantially more effective than others, however, and it has been found that as little as 0.02% of a polyvinyl ester made from commercial grade stearic acid is adequate to produce the desired crystal structure. This is the most active promoting material which has been investigated. The maximum amount of promoter which it is practical to use is one per cent. Over this amount product contamination results and the process is rendered unnecessarily costly. It is, of course, preferred in any operation to utilize as little modifier as is required.

The process of this invention will be more clearly understood by reference to the drawing which is a process flow diagram of the system of equipment. The fatty acid or fatty triglyceride to be separated is fed to the system from storage 10 through heater 11. It next passes through a vacuum dryer 12 where moisture is removed through steam ejector 13. The crystal promoter from a source of supply indicated at 13a is mixed with the effluent from the drier and the mixture is cooled in exchanger 14 to a point just above the crystallization temperature of the material. The fatty acid or triglyceride is then admixed with solvent wash entering through line 16 from wash solvent receiver

17. Solvent line 15 is utilized only upon start up of the plant. At this stage the stream consists of mixed fatty acid or triglyceride and crystal promoter all dissolved in solvent. This stream passes through line 18 into a scraped surface heat exchanger 20 of the type sold under the Trade Mark "VOTATOR" by the Girdler Process Equipment Division, Louisville, Kentucky. In this heat exchanger the solution is rapidly chilled in less than 1 minute through a range of approximately 75° F. to a temperature at which the saturated fatty material precipitates in the desired sand-like crystal form. Precipitation occurs in the heat exchanger 20 and in line 21 which extends to rotary drum filter 22.

The filter 22 is a conventional enclosed suction filter, and the cake is picked upon on the rotating filter drum on which it is washed by solvent flowing through line 23. The filter is of the type where the wash solvent is separately collected from the liquid fraction and the wash solvent passes through line 24 to wash solvent receiver 17. The filtrate which contains approximately one half of the original fatty acid or triglyceride passes through separate line 25 to filtrate receiver 26. Both of these receivers are connected by vacuum line 27 to vacuum pump 28 in order to provide the necessary suction on filter 22. It has been noted that the wash solvent from the wash solvent receiver is recycled to the fatty acid or triglyceride feed line through re-cycle conduit 16.

The filter cake which comprises approximately 50% of the mixed fatty acid or triglyceride feed is conducted to cake receiver 30 as indicated by line 31 where it is melted by steam coils 32. The melted cake passes from cake receiver 30 through conduit 33 to cake evaporator 34. In this evaporator solvent is evolved which is conducted through conduits 35 to condensers 36 and 37. From cake evaporator 34 the molten cake passes through line 39, heat exchanger 38, to cake stripper 40. Dilute solvent emerging from the top of this stripper passes through line 41 to an intermediate point in distillation column 42. The effluent solvent from the top of this column is conducted into line 35 extending to the condensers 36 and 37 by means of conduit 43. The bottoms from distillation column 42 comprising primarily water and some fat are conducted through line 44 to a fat separator and sewer not shown. The distillation column is heated by a conventional pot still arrangement indicated at 45.

The liquid fraction from filter 22 is conducted through line 25 to filtrate receiver 26

and from the bottom of this receiver through line 51 through solvent condenser 37. On passage through this condenser the filtrate is heated and following passage through condenser 37 flows through line 52 through heater 53 to filtrate evaporator 54. In this evaporator solvent is evolved as a vapor and passes through lines 55 and 35 to the solvent condensers 36 and 37. The condensed solvent from these condensers is conducted through line 56 to the solvent feed tank indicated at 57. From this tank solvent is re-supplied to the system through the cooler 58. The liquid fraction from the filtrate evaporator 54 passes out of the evaporator at the bottom through line 60, through heater 61 and into filtrate stripper 62. From the bottom of this stripper the liquid fraction is conducted through line 63 to storage 64. The solid fraction in liquid form is conducted from the bottom of cake stripper 40 through line 65 to solid fraction storage 66.

Each of the evaporators 34 and 54 is operated in such fashion that the feed is introduced near the top of the evaporator with a part of the product being recycled and intermixed with the feed, through the associated heater 53 in the case of the filtrate evaporator, and through associated heater 67 in the case of the cake evaporator. The solvent condensers 36 and 37 are cooled by water introduced through line 68 in the conventional manner.

Certain of the following specific examples demonstrate separations which may be effected utilizing a system of apparatus similar to that described above and will further illustrate the novel process of this invention.

EXAMPLE I

One liter of fully hydrogenated tallow containing about 1% FFA was heated to 260°—280° C. for 30 minutes in a stainless steel beaker, in the presence of 3 to 4 grams of powdered iron. The mixture was agitated by a gentle stream of air. The viscosity increased noticeably and the color turned from pale yellow to reddish brown. About 1% fullers earth was added to aid in filtering out the iron and remove some pigments, and the filtered liquid was cooled. The resulting product was found to be effective in promoting the desired crystal habit in concentrations as low as 0.2% based upon the weight of the fatty material.

There is given in the following table various properties obtained by laboratory tests of the starting material and the final product:

	Raw Material	Final Product
Iodine Value (WIJS Method)	0.45	1.23
Thiocyanogen Value	4.1	6.1
Peroxide Value	NH	15.4
Unsaponifiable Matter	0.26%	1.26%
Capillary Melting Point	60.6° C.	145° C.
Approximate Molecular Weight	860	15879

The increased average molecular weight of the product indicates that an appreciable amount of glyceride polymerization, probably by an addition reaction, has occurred. It is these polymers which comprise the effective crystal promoter. Thus, it would be expected, and has been found, that larger quantities of the product from the operation given above are necessary to achieve the same results, than would be the case if relatively pure polymers are used.

Other air-blown polymerized saturated fats are operative as crystal promoters. For instance, fully hydrogenated cottonseed oil, beef tallow, fully hydrogenated beef tallow and fully hydrogenated lard, when air-blown at a temperature between about 250° C. and 280° C. in the presence of iron for about 30 minutes, are sufficiently polymerized to form substances containing 4 to 6 fatty acid ester groups per molecule. These substances are effective at 0.2% concentration, based on the weight of fat or fatty acid to be crystallized.

In addition to the foregoing preferred crystal promoter the following substances have been tested for their efficacy as crystal promoting additives and found to be satisfactory:

A. Polyvinyl esters of the following: stearic acid, mixed hydrogenated soybean oil and unhydrogenated soybean oil fatty acids, lard acids, coconut oil acids, acids of partially hydrogenated sardine oil. Polyvinyl esters of saturated fatty acids having from about eleven to over 700 repeating vinyl units, with saturated fatty acid radical chains attached at essentially all such units have been tested. It is noteworthy that the effectiveness of these esters over such a wide range of "kernel" sizes supports the observation that the size of this "central kernel" is of little consequence.

B. Methyl glucoside stearate obtained by reacting methyl glucoside with twice the equivalent quantity of stearic acid. Methyl glucoside is a cyclic compound with four esterifiable hydroxyl groups, all of which are esterified by stearic acid in methyl glucoside stearate.

C. Palmitic esters of Hyprose SP80. Hyprose SP80 is a polyhydric alcohol which is derived from sugar and is believed to be 2-hydroxypropylsucrose, a polyalcohol with eight secondary hydroxyl groups. The palmitic ester was prepared by reaction of

Hyprose SP 80 in pyridine solution with an excess of palmitoyl chloride.

D. Aluminum stearate, which normally contains but three stearic acid groups but which when suspended in oil forms a fibrous micelle structure similar to polymerized material and accordingly contains numerous groups, also appears to exhibit the desired crystal promotion properties.

E. Sucrose esters such as sucrose stearate and the sucrose ester of soybean fatty acids.

EXAMPLE 2

A mixture containing 40 parts of distilled tallow fatty acids, having an iodine value of 50, and 56 parts of acetone, together with 4 parts of anhydrous methanol was prepared and heated to a temperature of 100° F. to effect complete solution of the tallow fatty acids in the mixed solvent. The ratio of solvent to fatty material was 1-1/2 to 1. The solution was pumped through a Votator scraped surface heat exchanger at a rate of 75 pounds per hour and was cooled by indirect heat exchange with liquid ammonia to a temperature of 4° F. The rate of cooling was such that the total residence time in the Votator heat exchanger was approximately 45 seconds. The slurry emerging from the Votator heat exchanger was extremely viscous and slimy in nature. Attempts to separate the solid and liquid phases of the slurry by filtration were not successful.

EXAMPLE 3

A mixture identical with that described in Example 2 was prepared and heated to 100° F. to effect complete solution. To 100 parts of the solution there was added three tenths part of a mixture consisting of 30% of the crystal modifier, prepared according to Example 1, and 70% tallow acids. The added constituents dissolved immediately in the hot solution. The resulting solution was pumped through a Votator heat exchanger at a rate of 75 pounds per hour and was chilled to a temperature of 0° F. during the 45 seconds residence time. The emerging slurry was found to be relatively non-viscous and was readily filterable. The slurry was filtered, and the filter cake was washed with solvent consisting of acetone containing 10% methanol. The filter cake was freed of solvent by evaporation and the resulting saturated fatty acid fraction was found to

have an iodine value of 5.2. The filtrate was freed of solvent likewise by evaporation and contained an unsaturated fraction having an iodine value of 93.5.

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EXAMPLE 4

There was continuously supplied to a system of apparatus similar to that shown in the drawing distilled tallow fatty acids having an iodine value of 72.1. These acids were pumped at a rate of 74 pounds per hour and were admixed continuously with crystal promoter, made in accordance with Example 1, which entered the system of apparatus at a rate of 0.11 pounds per hour. The fatty acids and crystal modifier were heated to a temperature of approximately 120° F. and were then contacted with acetone supplied at a rate of 143 pounds per hour. Thus the ratio of solvent to fatty material was 2 to 1, and it was found that the fatty acids and crystal promoter were completely dissolved in the acetone which had a temperature of 55° F. The resulting solution of fatty acids in solvent was found to have a temperature of 77° F. and at this temperature was introduced into the Votator heat exchanger where it was rapidly cooled to a temperature of about 15° F.

The slurry emerging from the Votator chiller was filtered and washed with acetone upon a rotary drum filter and thereby separated into two fractions. The saturated fraction was found to have an iodine value of 5.8 and constituted 50.4% of the original tallow acids. The unsaturated fraction was found to have an iodine value of 95.8 and constituted 49.6% of the original acids. The slurry of this example was very fluid and readily filterable.

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EXAMPLE 5

There was continuously supplied to a system of apparatus similar to that shown in the drawing a natural lard having an iodine value of 70. This lard was pumped at a rate of 72.5 pounds per hour and was admixed with 0.2% of crystal promoter, made in accordance with Example 1, introduced at a rate of 0.14 pounds per hour. The mixture of lard and modifier was heated to a temperature of 100° F. and was admixed with acetone at a temperature of 75° F. and supplied at a rate of 128 pounds per hour. The ratio of the solvent to lard feed was 1.75 to 1, and the resulting solvent lard solution had a temperature of 84° F. This solution was pumped through the chiller and emerged at a temperature of 28° F. From the chiller the resulting slurry was conducted to a filter, but the filter cake was not washed as in the previous examples. The filter cake was removed from the filter at a rate of 52.5 (on a solvent-free basis) pounds per hour and constituted 72.4%

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of the feed. The iodine value of the filter cake was 62. The filtrate was collected at a rate of 20 pounds per hour (on a solvent-free basis) and constituted 27.6% of the feed. The iodine value of this relatively unsaturated fraction was found to be 82. Solvent was removed from both the filter cake and the filtrate in the same manner as in the preceding example. To determine the suitability of the unsaturated filtrate fraction for use as a salad oil it was subjected to a cold test at 32° F. and failed to demonstrate clouding or precipitation for 2-1/4 hours.

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EXAMPLE 6

One kilo of stearic acid from hydrogenated soybean oil was dissolved in 2 liters of a mixture of acetone and anhydrous methanol (9 volumes to 1 volume). Two grams of polyvinyl stearate was dissolved in the solution. The polyvinyl stearate had been produced by ester interchange between polyvinyl acetate and methoxyethyl stearate and contained about 700 ester units per molecule. The polyvinyl stearate contained approximately 60 moles of stearic acid to 40 moles of acetic acid after the ester interchange. The fatty acid used in producing the ester was approximately 88% stearic acid, 10% palmitic acid, 1% arachidic acid and 1% oleic acid.

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The resulting solution was heated to 60° C. to ensure complete solution, then cooled to 50° C. in the air, followed by cooling with stirring in a water bath kept at 15° C. After the mixture reached 25° C. it was chilled in an ice bath until the temperature reached 5° C. At 19.2° C. the solution became cloudy and crystallization occurred. The grainy precipitate of stearic acid was collected on a filter, pressed and allowed to dry in the air. The mixture was more fluid and grainy than when the polyvinyl stearate had not been added. Without the additive the mixture was a thick slurry with a characteristic pearly sheen.

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EXAMPLE 7

One kilo of commercial saturated fatty acid dissolved in 2 liters of a mixture of acetone (9 volumes) and anhydrous methanol (1 volume) was treated with 0.2 gram (0.02%) of polyvinyl palmitate stearate made by acidolysis of polyvinyl acetate by commercial fatty acid containing 52% palmitic acid, 47% stearic acid and 1% oleic acid. Approximately 68% of the acetate radicals of the polyvinyl acetate were replaced by fatty acid radicals during the acidolysis to produce the polyvinyl palmitate stearate. The solution was heated to 60° C. to obtain complete solution, then cooled to 50° C. in the air. The mixture was stirred and cooled in a water bath maintained at 15° C. until the temperature of the solution reached 25°

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C. The mixture was then chilled with ice water until the temperature of the mixture reached 5° C. A clouding temperature of 17.6° C. was observed and a granular precipitate of saturated fatty acids was obtained which was easy to remove from the fluid mixture by filtration and pressing. Without the additive the mixture was very viscous and formed a soapy slurry having a characteristic pearly sheen.

The polyvinyl acetate used in this experiment was of low viscosity having an average of between 20 and 60 repeating vinyl units per molecule and the product of mixed polyvinyl fatty acid esters had an average molecular weight of approximately 15,000.

EXAMPLE 8

A mixture of 9 parts by weight of commercial triple pressed stearic acid and 1 part by weight of polyvinyl stearate (approximate molecular weight 200,000) was melted, thoroughly mixed and chilled. The solid product was converted into flakes for use as a crystal promoting additive.

One kilo of mixed acids obtained from a mixture of hydrogenated and unhydrogenated soybean oils was dissolved in 2 liters of a mixture of acetone (9 volumes) and anhydrous methanol (1 volume). To the solution was added 20 grams of flakes of polyvinyl stearate dissolved in stearic acid. The solution was then heated to 60° C., allowed to cool at 50° C. at room temperature, then chilled with agitation in a water bath at 15° C. The mixture became cloudy at 17.7° C. It was then placed in an ice bath and cooled to 5° C. The crystalline granular precipitate of stearic acid was removed by filtration, pressed and dried. A similar run without the additive of polyvinyl stearate gave a highly viscous slurry from which the stearic acid was very difficult to remove by filtration.

EXAMPLE 9

A. A mixture of 1 part of polyvinyl stearate in 9 parts of saturated cottonseed oil was melted, agitated until homogenous and chilled. This product was converted into flakes which were effective as crystallization promoters for saturated fatty acids.

B. One part of aluminium stearate admixed with 19 parts of commercial triple pressed stearic acid was melted, agitated until homogenous, chilled and converted into flakes. These flakes were effective as crystallization promoters in solid fatty acids.

EXAMPLE 10

To a solution of one kilo of tallow fatty acids dissolved in two liters of a mixture of acetone and anhydrous methanol (9 volumes: 1 volume) was added 2 grams of a polyamide resin produced from dimerized and trimer-

ized methyl esters of linseed oil fatty acids with an equivalent amount of ethylenediamine. This polyamide resin was produced by the method of Cowan et al *Oil & Soap*, April, 1944, pp. 101-107. The resulting solution was heated to 60° C. to ensure complete solution, then cooled to 50° C. in the air followed by cooling with stirring in a water bath kept at 15° C. After the solution reached 25° C., it was chilled in an ice bath until the temperature reached about 5° C. At 19.2° C., the solution became cloudy and crystallization began. A grainy precipitate of stearic acid was easily filtered on a filter press and dried in the air.

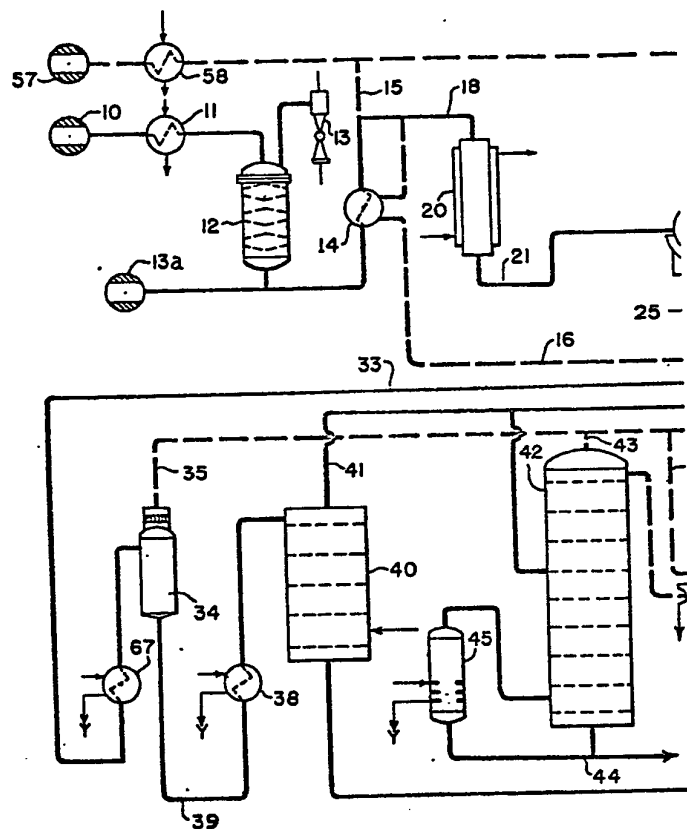
The same procedure with the same quantities of materials but without the polyamide resin led to a slimy precipitate of stearic acid in a viscous suspension which was very difficult to filter and afforded low purity stearic acid.

WHAT WE CLAIM IS:—

1. The process of separating mixtures of relatively saturated and relatively unsaturated fatty acids or triglycerides into saturated and unsaturated fractions by crystallization, the process comprising forming a solution of the fatty mixture containing up to 1% of a crystal promoter in the form of a compound having a nucleus or aliphatic chain to which are attached from 4 to 700 saturated or partially saturated fatty acid radical chains each chain having from 16 to 22 carbon atoms, the crystal promoter to the amount employed being soluble in the fatty mixture solution, chilling the solution of fatty mixture and crystal promoter to such a low temperature that the more saturated components of the mixture are precipitated in the form of discrete, non-slimy, readily filterable crystals, and separating the crystals from the mother liquor.
2. The process of claim 1 in which the fatty acid chains in the crystal promoter are more than 15% saturated.
3. The process of claim 1 or 2 in which the ratio by weight of the solvent to the fatty mixture is within the range of from 1 to 1 and 2 to 1.
4. The process of claim 1, 2 or 3 in which the step of chilling the composite solution to effect precipitation of the more saturated components of the mixture is effected in less than one minute.
5. The process of any of claims 1 to 4 in which the crystal promoter is a polyvinyl ester of a saturated fatty acid.
6. The process of any of claims 1 to 4 in which the crystal promoter is methyl glucoside tetrastearate.
7. The process of any of claims 1 to 4 in which the crystal promoter is a palmitic ester of a polyhydric alcohol.

8. The process of any of claims 1 to 4 in which the crystal promoter is aluminum stearate in an oil suspension.
- 5 9. The process of any of claims 1 to 4 in which the crystal promoter is a sucrose ester of a saturated fatty acid.
- 10 10. The process of any of claims 1 to 4 in which the crystal promoter is a polymerized triglyceride composition obtained by heating the triglyceride to a temperature in the range of from 260° to 280° F. in the presence of air.
- 15 11. The process of any of claims 1 to 4 in which the crystal promoter is a polyamide resin formed by condensation of dimerized or trimerized vegetable fatty acids with an aliphatic or aromatic polyamine.
- 20 12. The process of claim 1 for separating mixtures of relatively saturated and relatively unsaturated fatty acids or triglycerides substantially as described in any of Examples 1, 3 to 8 and 10.
13. A crystal promoter for saturated fatty acids which comprises an ester of a polyhydric alcohol containing at least 4 fatty acid groups per molecule wherein the fatty acids contain 6 to 22 carbon atoms suspended in an oil or fat.
- 25 14. A crystal promoter for crystallization of solid fatty acids which comprises an aluminum salt of a fatty acid containing 16 to 22 carbon atoms in fibrous micelle structure suspended in an oil or fat.
- 30 15. A crystal promoter for crystallization of solid fatty acids when prepared substantially as described in any of Examples 1, 6, 8, 9 and 10.
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COMPLETE SPECIFICATION

1 SHEET

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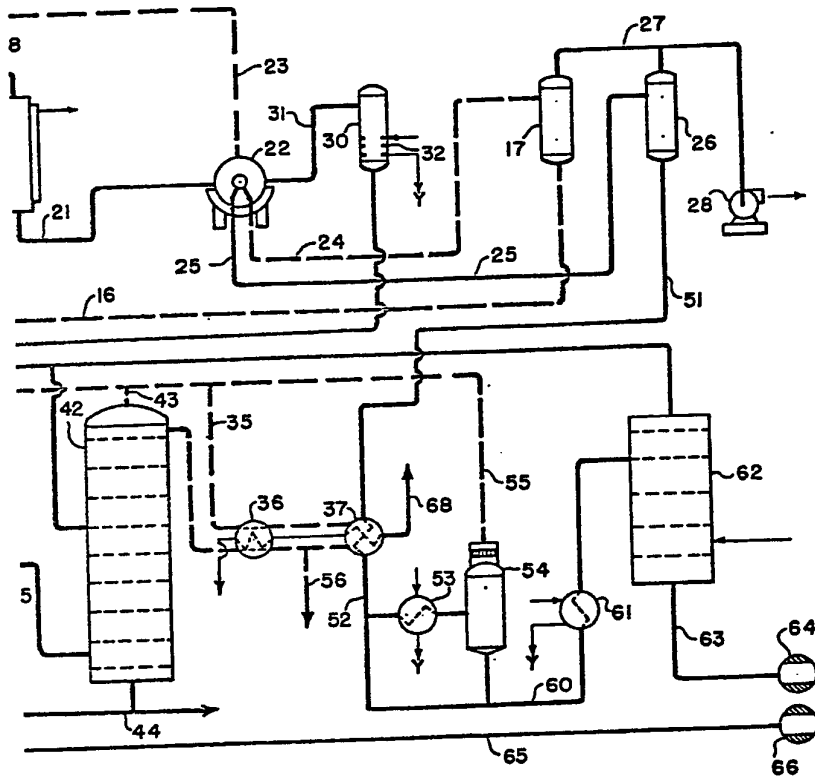


Fig. 1

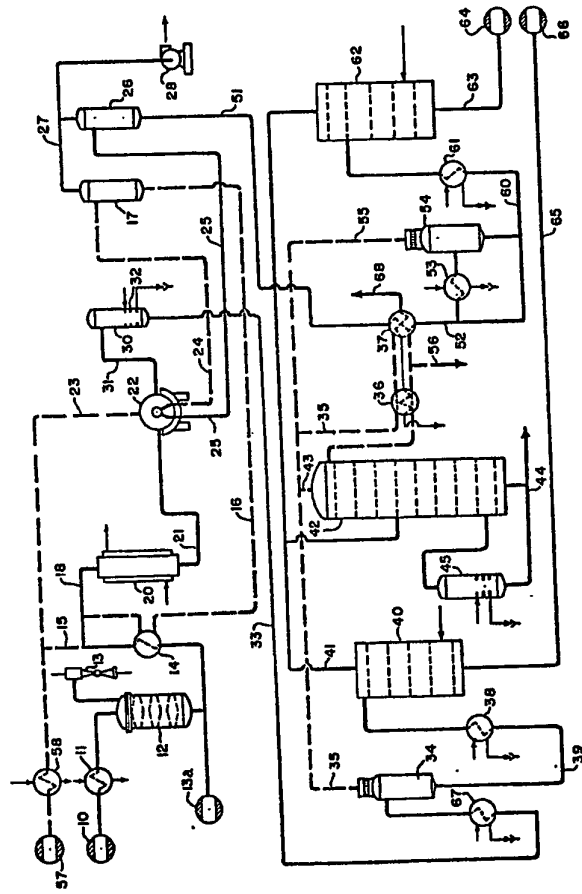


Fig.1